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Methods for Manufacturing Acid-Precipitated Casein from Skim Milk

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INTRODUCTION

Since 1930, more than 100 billion pounds of milk has been produced annually in the United States. More than half of the milk is separated on the farm or in the factory to provide the cream used in making butter and ice cream, and for bottling, with the result that more than 50 billion pounds of skim milk is produced annually as a by-product.

Although it has a high and unique nutritive value, only about 10 percent of the skim milk supply is normally used as human food. Another 3 percent is used as a source of commercial casein, and the

[1] This is a revision of former editions (1933 and 1938) that were prepared by Charles S. Trimble, associate dairy manufacturing specialist, and R. W. Bell, senior dairy manufacturing specialist, Bureau of Dairy Industry. In the current edition, liberal use is made of extracts from the 1938 edition, in the sections Establishing a Plant, Methods of Manufacture, and Marketing.

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balance—over 40 billion pounds—is either fed on the farm or utilized by commercial companies in making animal feeds.

Some of the skim milk retained on the farm for feeding hogs, calves, and poultry would be displaced with other feeds if farmers were able to obtain sufficient returns for the skim milk to make it profitable to change to other feeds and to the delivery of whole milk instead of cream.

The manufacture of dry skim milk for human food is increasing at present as a result of Government purchases for wartime uses, and there apparently is need for still larger quantities than are yet reaching the drying plants. While it would be highly desirable from a national health standpoint to utilize all the skim milk for human consumption, much of it is produced in areas remote from established drying plants and where it often would not be feasible or profitable to operate a skim-milk drying plant. In such areas, however, it would usually be possible to equip the local dairy plant or creamery with the few facilities needed to make wet casein. Thus, skim milk not needed for feeding farm animals, and not available to manufacturers of dry skim milk for human consumption, could be made available for making casein.

Casein of uniform high quality is required in most industries consuming this product. The paper-coating industry, which consumes over three-fourths of the domestic casein produced, is especially insistent on high quality and it is desirable and profitable, therefore, for the manufacturer of casein to use special care in its processing.

No special skill is required to make casein and, though expensive labor-saving and time-saving machinery may be employed, a product of the highest grade can be made with simple and inexpensive equipment. However, a knowledge of the characteristics of high-grade casein and an understanding of the procedures necessary in order to obtain such casein are essential.

This circular contains information of practical value, both to present manufacturers who wish to improve their product and to prospective manufacturers who wish to make the best grade of casein possible. High quality of product is stressed and economy of operation is kept in mind throughout. A list of necessary equipment is given.[2] Reasons for the successive steps of the process are discussed, and specific procedures are outlined. The more important methods of testing and analysis are described in detail. This information should make it possible to establish a plant for making acid-precipitated casein and to make consistently a high-grade product.

PRODUCTION AND CONSUMPTION IN THE UNITED STATES

With the exception of relatively small quantities of casein precipitated by rennet for use in plastics manufacture and in foods, practically all domestic casein is produced by using lactic, sulfuric, or hydrochloric acid as the precipitating agent. Approximately half of the domestic acid-precipitated casein is manufactured in Wisconsin,

[2] The author recognizes that supplies of some of the materials and equipment mentioned throughout the text are not readily available because of wartime priority ratings. Substitutes are mentioned wherever it seemed of value to do so, but because of the rapidly changing situation definite recommendations could not be made in all cases.

Illinois, and Minnesota. These 3 States and California, Idaho, New York, and Vermont produced over 90 percent of the total domestic output for the years 1937 to 1940, inclusive. The number of plants producing casein varies considerably from year to year, as shown in table 1, but this is not a reliable index of the quantity produced, since many dairy plants operate their casein equipment only a few months of the year, and, in years when prices of cheese, condensed skim milk, or milk powder are relatively high, they may not make any casein. It is evident, therefore, that considerable increase in production is possible without an increase in the number of plants, provided that these plants are operated a greater portion of the year than has been customary in the past.

TABLE 1.—*Number of plants manufacturing casein and quantity manufactured in the United States, quantity imported, and domestic market, in the 12-year period 1929–40* [1]

Year	Domestic production		Quantity imported	Domestic market (production plus imports	Year	Domestic production		Quantity imported	Domestic market (production plus imports)
	Plants	Quantity				Plants	Quantity		
	Number	*Pounds*	*Pounds*	*Pounds*		*Number*	*Pounds*	*Pounds*	*Pounds*
1929	214	30, 537, 000	27, 583, 000	58, 120, 000	1935	340	37, 638, 000	3, 230, 000	40, 868, 000
1930	316	41, 965, 000	18, 499, 000	60, 464, 000	1936	552	46, 140, 000	16, 209, 000	62, 349, 000
1931	222	35, 335, 000	3, 503, 000	38, 838, 000	1937	624	67, 467, 000	5, 210, 000	72, 677, 000
1932	195	24, 428, 000	1, 201, 000	25, 629, 000	1938	566	48, 549, 000	417, 360	48, 966, 360
1933	244	24, 087, 000	8, 142, 000	32, 229, 000	1939	315	40, 878, 000	17, 536, 000	57, 852, 000
1934	368	37, 331, 000	1, 491, 000	38, 822, 000	1940	[2] 210	[2] 48,037, 000	24, 523, 000	[2] 72,560, 000

[1] From statistics compiled by the Bureau of Agricultural Economics and the Agricultural Marketing Service.
[2] Preliminary figure.

Statistics on production and importation of casein are given in table 1. After the increase in the tariff rate from 2.5 to 5.5 cents per pound, effective June 17, 1930, imports declined sharply. Consumption decreased at about the same time and remained fairly constant until 1936. At that time consumption increased to the earlier level, this increase being met by increased domestic production, since low prices and the tariff barrier acted to prevent any considerable amount of importation for several years. Higher prices and the loss of the European market for casein again caused imported casein to become a factor in the domestic market in 1939 and 1940.

Effective November 15, 1941, the tariff was lowered from 5.5 to 2.75 cents per pound. At that time it was being urged that as much domestic skim milk as possible be converted into evaporated and dry skim milk for food for Great Britain.

There is no apparent reason why present uses should require any greatly increased quantity of casein. The only factor at present tending to increase the demand for casein is its use in the manufacture of textile fiber. Efforts are being made to substitute soybean protein for casein in paper coating, in glue making, and in the manufacture of fiber and, if successful, will tend to decrease the demand for casein.

ESTABLISHING A PLANT

When conditions are such that there is an oversupply of food products made from skim milk and prices for these products are low, the

diversion of skim milk into casein may be of material benefit in stabilizing and strengthening markets for other skim-milk products. In sections where such food products cannot be made or marketed readily, the manufacture of good-quality casein should be given serious consideration as a means of utilizing skim milk to advantage. In many cases, also, it is not advisable to equip small dairy-products plants with expensive condensing or desiccating machinery when casein equipment can be installed at a relatively small cost. In a plant equipped for making food products from skim milk, the special equipment needed for producing casein is comparatively inexpensive and increases the flexibility of operation. It is often advisable to arrange equipment and operations so that the dairy-products plant can be changed quickly from the manufacture of one product to another without materially affecting the operating efficiency. The more flexible the dairy-products plant, the easier it is to take advantage of fluctuating market conditions.

Casein can be made during any season of the year. When ordinary care is exercised in storing the finished product, it can be kept for months without deterioration. Therefore, casein can be made in isolated sections because the distance from market is not as important as in the marketing of perishable food products.

Volume and Quality of Skim Milk Required

The volume of skim milk necessary for successful operation of a casein factory is greater when casein is the only product made than when other skim-milk products are made also. If casein is the only product to be made, it will probably be necessary to process at least 25,000 pounds of skim milk daily, and even larger quantities are advisable. The cost of manufacture per unit amount of casein should decrease with an increase in the daily volume of business. When other products are made in a plant that is equipped to make casein, then casein can be made from a small daily volume of skim milk at very little additional cost for labor and power.

Sweet, clean, unpasteurized skim milk, fresh from the separator, is desirable if high-grade uniform casein is to be made. Such factors as fermentation, and excessive fat and foam in the skim milk, may deleteriously affect the quality of the finished product.

Location of Plant

It is practical to make casein in small creameries in which only a few thousand pounds of skim milk is produced daily, provided a central drying plant is available at a moderate distance from each of a group of producers of casein. Ordinary creamery equipment can be used for the most part, a home-made curd press being the only additional item that is absolutely essential. It is highly important that the curd be delivered to the drying plant promptly after pressing. One convenient arrangement is for a centrally located creamery to install a drier of sufficient capacity to dry the wet casein produced by a number of nearby creameries.

If the volume of skim milk available is large enough to justify the establishment of a casein factory, the location of the building

should be given careful consideration. A separate building is advisable for large-scale production. This building should be convenient to the main plant to facilitate transferring the skim milk and supplying the power, the steam for heating and drying, and the large amount of water required in the washing process. Adequate facilities for sewage disposal should be available, as well as facilities for shipping the casein and receiving coal and other supplies. When the amount of casein to be made does not warrant the erection of a separate building, the space allotted to casein manufacture should be separated from other departments as much as possible and be ample for all operations.

Equipment—Batch Process

Practically the same equipment is needed in making any type of casein by the batch process. This equipment is neither elaborate nor expensive. Some of it is regular dairy equipment and some of it may be constructed at the plant. If the casein equipment is to be used only a few months of the year, it is especially important that both initial cost and depreciation be kept at as low a value as possible.

The equipment listed below is sufficient for processing 10,000 pounds of skim milk daily:

One 1,000-gallon tank for storing skim milk.
Two 5,000-pound (or one 10,000-pound) milk or cheese vats, or unlined wooden vats, for holding the skim milk for precipitating and washing the curd.
One 1,000-gallon tank for storing whey when lactic casein is made.
Facilities for determining acid or bromocresol green indicator paper.
Two 2-inch centrifugal pumps for skim milk and whey.
One or two curd presses with dividing boards and press cloths.
One curd mill of the shredder-beater type.
One hundred and eighty standard drying trays, 30 inches square, with 8- to 10-mesh galvanized-iron screening.
Six tray trucks.
One drying tunnel 22 feet long, 5 feet high, and 32 inches wide.
One high-speed fan, 30 to 35 inches in diameter, equipped with motor for circulation of heated air.
Radiators or steam coils for drier, together with steam trap for condensation.
One recording thermometer for use in control of temperature of air in drier.
One grinder.
Miscellaneous small equipment, such as strainers, pulleys, steam piping, valves, steam and water hose, rakes or paddles, thermometers, pails, bags, and scales for use in weighing dried casein.

More vat space will ordinarily be required in making lactic casein than in making other acid-precipitated caseins, and extra containers will be required for holding the whey to be used for starter. In making hydrochloric and sulfuric casein, containers in which to dilute the sulfuric or hydrochloric acid will be needed.

If wooden vats instead of metal cheese vats are used, often such vats may be constructed at the casein factory at less cost than when purchased as complete units. Redwood, cypress, pine, or other suitable wood can be used. The vats should be made of 2-inch stock and should be braced with rods or long bolts under the bottom and along the sides. The bottom should slope toward the outlet to facilitate drainage. Steam lines for delivering steam for heating the skim milk should be installed, either along the sides of the vat or overhead. Open vats in

which direct steam is admitted are commonly used in making casein, although jacketed vats afford better control of temperatures.

The tanks for storing the skim milk and whey should be placed where they can be kept clean and sanitary. Either metal or wooden vats may be used.

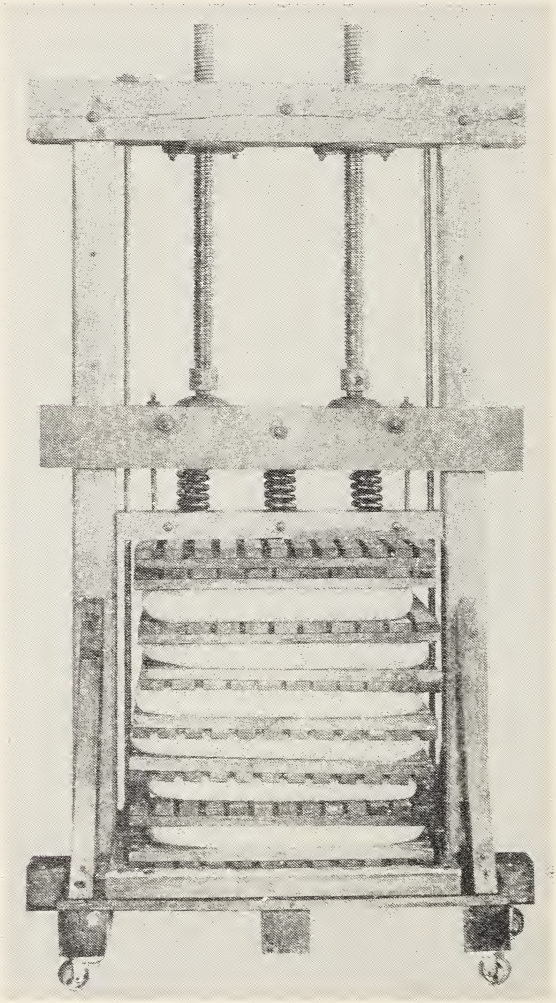

FIGURE 1.—Casein curd press (batch type).

The curd presses (fig. 1) should be constructed of heavy material, so that considerable pressure can be applied to the wet curd. It is cheaper to expel moisture mechanically than to vaporize it by means of heat. A properly pressed curd mills and spreads more easily than a poorly pressed curd. Special mechanical presses (fig. 2) are now available for large-scale production. When the quantity of casein made justifies the extra investment, these special presses save much time and labor.

The pressing of the curd then becomes a continuous process. Their operation is so effective that the pressed curd has a moisture content of approximately 50 percent.

Burlap-sacking material is commonly used as press cloths in casein plants, but its use for this purpose is not advisable, as it is hard to clean and keep clean. A cloth that is somewhat heavier and closer woven than burlap is more satisfactory. Heavy white duck (No. 10 size), lightweight canvas, or similar cloth material will give better service than burlap. Some companies make a specially woven heavy

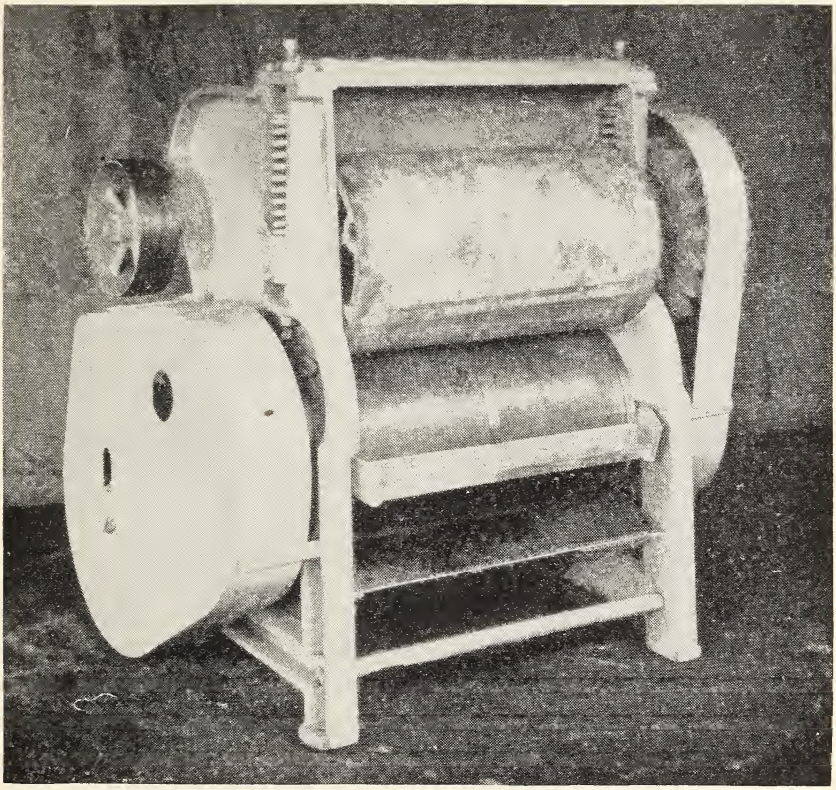

FIGURE 2.—Casein curd press (continuous type). Courtesy Ireland Machinery and Foundry Co.

cloth which can be obtained in any size desired and is very satisfactory.

A shredder-type curd mill will give good service except when cooked-curd casein is made.

It is important that galvanized screening be used on the drying trays as ordinary screening soon rusts and small metal particles get into the finished product. Copper screening is durable but will often impart a green color to the dried casein. During the slack season of production the screens should be shellacked or varnished to preserve them and lengthen their life.

A tunnel of the dimensions given for a 10,000-pound plant is large enough for drying the casein from 15,000 pounds of skim milk. A

drying tunnel (fig. 3) of this size is more satisfactory than a smaller one, since it allows for some increase in quantity of milk processed. It is often desirable from the standpoint of economy of space and construction to have two tunnel driers built together, with one wall in common. The tunnel or tunnels should be located where they will be convenient and room should be provided for installing additional tunnel units if the volume of business makes this necessary in the future.

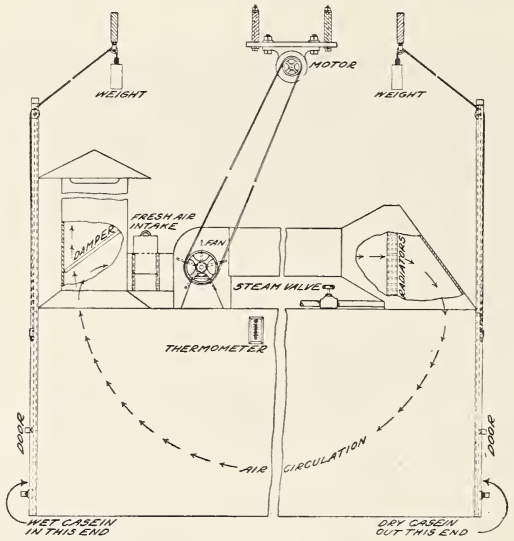

FIGURE 3.—Casein drier (tunnel batch type).

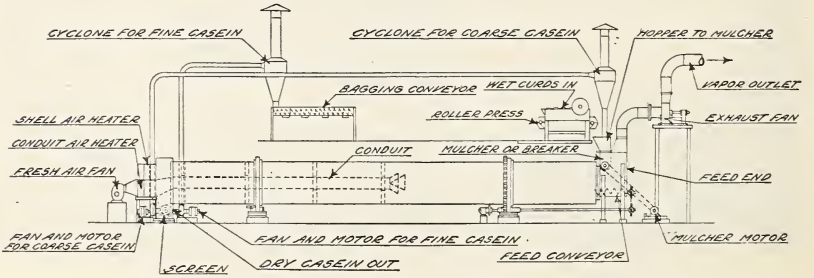

FIGURE 4.—Casein drier (revolving-drum continuous type). Courtesy Louisville Drying Machine Co.

The most advantageous place for the installation of the tunnel in a two-story building is usually on the second floor. A continuous drier suitable for large plants is shown in figure 4.

The drying of casein is the largest single cost item in the manufacturing process. An improperly designed and constructed drying unit is expensive to operate, as it requires too much power and heat and takes too long to dry the casein properly, and the quality of the finished product may be deleteriously affected. The construction of the drier, therefore, should be given careful attention, for a properly constructed drier will, because of savings effected, be the most eco-

nomical in the end. The drier should be not only airtight in construction, but also insulated to conserve and retain heat. The inside of the tunnel should preferably be lined with metal rather than with wood. The steam pipes leading to the heating coils should be insulated. There should be ample room at both ends of the tunnel to facilitate handling the trucks in filling and emptying the drier.

Many of the drying tunnels now in operation are built 6 feet high, instead of 5 feet as given in the list of equipment. The added height is more convenient for handling the loaded trucks, and when space permits is desirable. Correct and controlled air circulation is an important factor in the production of high-grade casein. The fan should be of the high-speed type to insure efficient circulation of air. The floor of the room, if airtight, can be used as the floor of the tunnel. A recording thermometer will aid in safeguarding against too high a drying temperature.

On large-scale production it is advisable to have a grinder that can be adjusted to grind the dried casein into particles of any desired size. Either a burr-type or roller-type grinder may be used. With the roller type the casein passes through adjustable steel rolls, where it is ground to the desired fineness. The casein ground in such grinders is usually more uniform in particle size than that ground in a burr-type machine. The latter type, however, is generally somewhat cheaper in price and for a small plant will give satisfactory service. Magnetic separators that remove metallic material, such as nails, bolts, or bits of screen, that may have gotten into the casein, are used by some jobbers. While most of the casein marketed is ground to pass through a 20- to 30-mesh screen, some users require a more finely ground product, or one that will pass through an 80- to 100-mesh screen.

The grinder may often be omitted from the equipment, especially in small-scale manufacture. Some jobbers and users of casein even prefer to do their own grinding.

As with the wooden vats mentioned, it is often possible for a casein manufacturer to make other equipment at substantial savings. Trays and tray trucks, and often the drying tunnels may be so made at less expense than when purchased as complete units. It is advisable, however, to consult experienced casein manufacturers and dealers as to the best equipment to make or purchase. Many of the manufacturers, dealers, and dairy-supply companies are in a position to furnish valuable information on the proper equipment and its correct installation, and to solve the engineering problems involved, in order to obtain the most efficient operation.

As long as the precipitating and washing operations are carried out in vats (fig. 5) and the tunnel method of drying prevails, the equipment needed for either a small or a large plant will not vary much from that listed in page 5, other than in the size and number of units. It is true, no doubt, that the finished product made by special equipment and processes will be more uniform in quality than that made by the ordinary hand methods.

The drying should be done at the original plant whenever possible, since the wet curd deteriorates rapidly.

The equipment installed in the usual casein factory, while it need not be elaborate, should be constructed and arranged so that the

different units can be kept clean and sanitary. Clean equipment is an asset of material importance in the making of a quality product.

It is not possible to give cost figures for building and equipment that would apply to all sections or local conditions.

Estimates on prices of equipment received from casein dealers and jobbers of casein equipment vary within rather wide ranges. The estimated price of equipment necessary for processing 5,000 to 10,000 pounds of skim milk daily ranged from $800 to $1,500 f. o. b. origin. This wide range is due to differences in quality and kind of equipment listed. For example, wooden vats may be purchased or built for from $100 to $150 each, while a metal cheese vat would cost from $300 to $350. Such vats would be much higher in price if they were equipped with mechanical agitators. Curd presses of the usual design range in price from $85 to $175, curd mills from $100 to $300, screen trays from 60 cents to $1.10 each, and complete drying tunnels from $400 to $800.

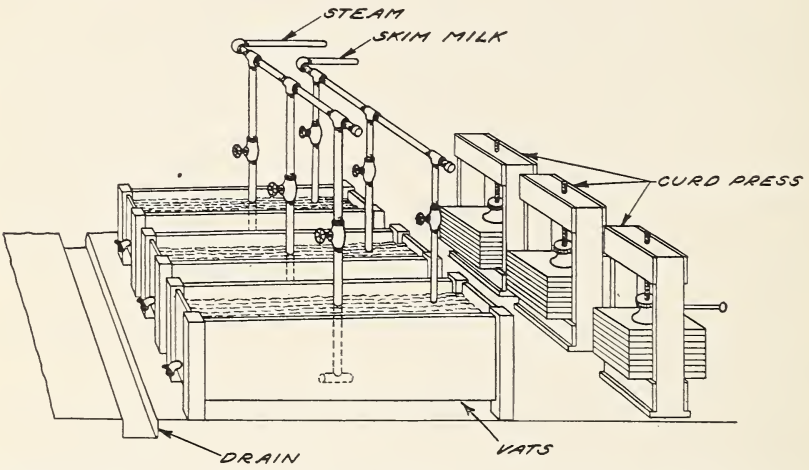

FIGURE 5.—The vat method requires a longer time and more expense for labor than the continuous process.

Cost estimates obtained from casein manufacturers also vary within rather extreme ranges. Some managers of plants in which 8,000 to 12,000 pounds of skim milk are processed daily placed the total cost of the equipment only at from $700 to $800. Others who process about the same amount of skim milk gave a figure materially higher, ranging from $1,200 to $1,500, and even more. These figures are all for factories which operate as branch departments of a milk-products plant, and do not include charges for transportation, installation, and building space in the main plant, or new construction. As already stated, the total cost of necessary equipment can often be reduced when a plant is able to build or have much of its equipment made locally. It is false economy, however, to attempt to manufacture casein with inadequate or poorly constructed equipment. Not only is operating efficiency impaired, thus raising manufacturing costs, but the quality of the finished product may also be harmed. Suffi-

cient capital should be provided to supply not only adequate equipment, but also to provide for its proper installation and arrangement in the plant.

On the basis of a survey in 1930 of a large number of casein factories operating in various sections of the country, the total cost of complete equipment and materials, and installation, exclusive of building space, varied from $1,500 to $2,500 for the smaller plants, and from $3,500 to $4,500 for the larger plants, depending upon the existing local conditions and market prices. These figures are only approximations, but indicate what the prospective manufacturer of casein would have to invest in order to start a casein factory in conjunction with a milk-products plant.

A separate building or an addition to an older building would, of course, add to the cost estimates given, and the prospective manufacturer would have to be governed by previous experience as to the building materials and the size and type of building best suited for local conditions and needs. Advice from reliable sources should be sought and when at all feasible, an inspection visit made to several efficiently operated casein factories before construction is started and equipment purchased or installed.

Equipment—Continuous Process

The equipment required for a continuous-process plant is much more expensive than that used in a batch-process plant, an investment of the order of $50,000 being necessary. Such a large investment is feasible only when a comparatively large quantity of skim milk is available throughout the year and when savings in time and labor cost are important. In locations where 100,000 pounds of skim milk are available per day, it will be practicable to establish a plant for manufacture by the continuous process. The necessary items include the following equipment:

Tanks for receiving and storing skim milk.
One 2-inch centrifugal pump for handling skim milk.
Tubular heater.
Machine for continuous precipitation.
Continuous curd press and mill.
Continuous drier, with conveying equipment for feeding milled curd and carrying dry casein to grinder.
Grinder, with screening and sacking equipment.
Scales for weighing dry casein.

Drying tunnels with necessary adjuncts may be used instead of the more costly continuous drier, provided their capacity is sufficient to receive the milled curd as fast as it is produced.

The Sheffield continuous machine (fig. 6) is an example of successful continuous equipment for precipitating casein. A two-roll continuous curd press is shown in figure 2. The machines shown and others for the same purposes are patented and cannot be used without payment of royalty.

MANUFACTURING

Acid-precipitated caseins are, as the name indicates, caseins that have been precipitated by acid and, for the most part, have the same

characteristics regardless of the acid used. The differences in caseins depend on such factors as temperature and degree of acidity during processing, amount of washing, and the care employed throughout the whole process of manufacture.

In considering methods of manufacture, caseins may be classified on the basis of the acid used; hence, lactic, sulfuric, and hydrochloric (muriatic) caseins. Another classification is based on the type of curd produced; hence, pressed-curd, cooked-curd, grain-curd, and continuous-process caseins. Any one of the three acids may be used in making curd of any type. Lactic caseins may be classified further into self-sour and direct-precipitation caseins, and cooked curds may be subdivided into cooked and semicooked curds.

Fundamental Considerations

Since high-quality casein is in the class of high-grade chemicals, the skim milk used in casein manufacture should be fresh, clean, and sweet, the equipment should be adequate and clean, and the operator should understand the reason for each step in the process, should be capable of making and judging the necessary control tests and of appreciating the need for scrupulous care in carrying out the operation.

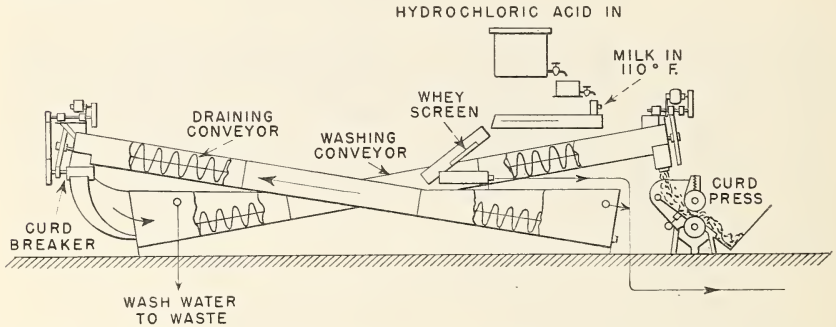

Figure 6.—Sheffield continuous casein precipitator. Courtesy Sheffield Farms Co., Byproducts Division.

Casein is present in cows' milk in about 3 percent by weight. It is combined with calcium and held in colloidal solution at approximately the neutral point (pH 6.6). If acid is added slowly to milk, casein precipitates as a very watery, gel-like curd, which can be made to expel much of its water either by adding more acid or by heating. The acid added to the milk combines with the calcium, leaving the casein free and insoluble. This reaction is sluggish and may be speeded both by an excess of acid and by heat.

Casein is least soluble at the reaction of pH 4.6, but, once out of solution, does not dissolve appreciably in either slightly more acid or slightly more alkaline solutions. Consequently, it is practical to add acid until the reaction is at pH 4.1, or even lower, without redissolving casein. This is done in the grain-curd and the continuous processes to hasten the removal of calcium and to improve the texture of the curd. Excessive heating causes the curd to be first stringy and plastic and later "rubbery," a condition rendering the curd difficult to wash and the casein of poor solubility in alkaline solvents.

After the casein is precipitated, it is necessary to get rid of the water, salts, and other soluble materials of the whey. After draining much whey still remains in the curd, which must then be washed several times with water to insure a casein as free of salts or ash as possible.

As much as possible of the water remaining in the washed curd is removed by means of pressure, this being less expensive than drying by means of heated air. Pressure reduces the moisture to approximately 55 percent. The curd must then be shredded and the removal of moisture completed promptly by means of a heated drier to avoid mold and bacterial decomposition. The dried casein is usually ground before being marketed.

General Procedure

PRECIPITATING

Degree of acidity, temperature, and rate of stirring regulate the characteristics of the curd obtained. When acid is being added to the skim milk, some stirring is necessary to distribute the acid. Rapid stirring at temperatures below 100° F., however, causes the particles of curd to be fine and slow to settle in draining and washing, and should be avoided. In self-sour processes stirring is needed to break up the curd so that it can settle.

Temperature should be controlled accurately, since curd is very sensitive to heat. In batch processes a water jacket on the vat is the most satisfactory means of temperature control; in continuous processes a preheater is adequate. Curd precipitated at a temperature below 90° F. is very soft and fine, and consequently is difficult to wash without loss. Curd formed at temperatures between 95° and 100° F. is coarse, provided stirring is not too fast. Such a curd can be made firm in either of two ways. One is by heating to a temperature between 100° and 118° F., the exact temperature depending on the acidity. The other is to increase the acidity to pH 4.1 without raising the temperature. This latter procedure produces a curd of the firm, granular type, known as grain curd, which is particularly easy to drain and wash thoroughly.

Curd precipitated at 110° F. has what is perhaps best described as a "chewing-gum" texture; it is stringy, lumpy, and coarse and there are practically no fine particles present. As this curd cools below 110° F., it is easily chopped into coarse grains very similar to grain curd in appearance and as readily washed free of salts. This is the type of curd made in the continuous processes.

Semicooked curd, precipitated at 130° to 135° F., and cooked curd, heated at 160° to 180° F., are rubberlike and plastic and very difficult to wash. The casein resulting from a cooked-curd process is hard, tough, difficult to dissolve in the usual casein solvents, and high in ash and free acid.

The acidity necessary for precipitation may be obtained by the growth of lactic-acid-producing bacteria in the skim milk, by the addition of sour whey, or the addition of sulfuric acid or hydrochloric acid. When vat space is ample and it is desired to save the cost of acid, the fermentation (self-sour) method of precipitation is suitable. In using this method, a vigorously growing whey starter is added to

the milk and several hours must then be allowed in the time schedule for development of the required quantity of acid in the milk. It is possible, however, by the growth of high-acid-producing lactobacilli, to obtain a sour whey containing 2 percent or more of lactic acid. Such a sour whey may be used for the direct precipitation of casein from skim milk, about one part of whey to three parts of skim milk being sufficient. In some plants, the self-sour method is employed to form the curd, the whey is drained off, and a very sour whey added to bring the acidity to the point where the texture of the curd is suitable for adequate washing.

When sulfuric or hydrochloric acid is used as the precipitant, it is necessary to dilute the commercial acid before adding it to the skim milk. In general, the more dilute the acid, the better the quality of the casein produced. Practically, sulfuric acid is used in dilutions of from 1 part in 6 parts of water to 1 in 30; or hydrochloric acid in dilutions of from 1 in 3 to 1 in 9. In diluting acid, earthenware containers should be employed, and great care must be taken because of the heat developed during dilution. If water is poured into sulfuric acid, hot acid will be spattered about and the operator is likely to suffer severe burns. Sulfuric acid should be poured slowly into the water with continuous stirring and the water should be cold. It is advisable for the operator to wear goggles and rubber gloves while diluting acid.

The acidity of the whey surrounding the curd may be expressed as percentage of acid. This is determined by titration with a standard alkali solution as in the determination of the acidity of milk, and is expressed as percentage of lactic acid regardless of the acid used. The acidity may be expressed also as pH value, which is determined with a color indicator, either as a solution or as indicator paper. The same percentages of different acids, even though calculated as the same acid, impart different intensities of sourness to milk or whey; hence, close correspondence of values obtained by the two methods cannot be expected. Even the same quantities of the same acid when added to different milks, may give wheys with different pH values.

Curd begins to be apparent in skim milk at a temperature of 100° F. when the pH value reaches 5.3, which value, if the acid present is lactic, corresponds to somewhat less than 0.5 percent of acid. At pH 4.8 to 5.0, or from 0.5 to 0.6 percent acid, the curd is so well defined that, by heating to 104°–106° F. and stirring, it will precipitate, leaving a clear whey. At pH 4.7 to 4.8, or from 0.55 to 0.65 percent acid, no heating is required and stirring for 10 to 15 minutes is all that is necessary to obtain complete precipitation and a clear whey. When a grain curd is made by the use of hydrochloric acid, the necessary pH value of 4.1 is attained at a titratable acidity of about 0.5 percent. When the acid is sulfuric and the temperature of precipitation is 120° to 125°, as little as 0.32 percent of titratable acidity is sufficient, but a very tough curd is obtained.

Low temperatures and weak acidities both tend to produce fine, soft, "mushy" curd that is very difficult to separate from its whey. High temperatures produce tough, rubbery curd from which it is practically impossible to wash impurities. By precipitation in the temperature range of 95° to 115° and the pH range 4.8 to 4.0, curd can be obtained that separates sharply from its whey and that may be

washed relatively free from the salts and other impurities present in the whey. A reaction of pH 4.1 and a temperature of 95° to 100° F. gives the most granular curd, which is easiest to wash. If the curd is to be chopped during draining, as in a continuous machine, the temperature may be raised to 110° to 115° and advantage thus taken of the expelling of whey that the higher temperature accomplishes.

Temperature should, of course, be controlled with the aid of thermometers. Acidity may be controlled by means of titration, or, still more precisely by means of indicator solutions or indicator papers. With experience, the operator can usually judge the acidity roughly by the color of the whey or by the "feel" of the curd when it is squeezed in the hand, but these tests are not recommended for use in place of titration or indicator tests. The technique of determining titratable acidity is familiar to all dairy workers and need not be described here. The indicator method is the quickest and most accurate method for controlling acidity and bromocresol green is the indicator most suitable in the range pH 4.0 to 5.0. Kits may be bought containing bromocresol green solution, color standards for different pH values, and directions for use. Indicator papers with paper standards are more convenient in a plant and are fully as satisfactory. For use, the paper is dipped in the whey, placed on a window pane not in sunlight and judged immediately. The color is a deep blue above pH 5.2 and changes through green to yellow as the acidity increases to pH 4.0. At pH 4.8 the color is a bluish green and at pH 4.1 a greenish yellow. With a little experience, values can be judged accurately even without a comparison of colors with standards.

DRAINING

Whey should be removed from casein curd as soon as possible after precipitation is complete and the curd has settled for a few minutes. The longer the curd remains in contact with the whey, the more difficult it will be to wash out acid, milk sugar, and salts. Furthermore, undesirable changes take place in the curd on standing in contact with whey.

In draining curd made by a batch process, especially if it is of the granular type, the curd is first pushed back from the outlet end of the vat and allowed to settle, as in cheese making. A strainer is then inserted in the gage valve from the inside, and the valve is opened full width. When a sloping bottom has not been built in the vat the outlet end of the vat should be lowered to facilitate drainage. A burlap sack tied over the gate valve will prevent the loss of small particles of curd. A coarse strainer of heavy wire or a board inserted in the vat about 3 or 4 feet from the outlet and extending across the vat, will also serve to hold the curd back and facilitate drainage.

WASHING

Effective washing of the curd is extremely important in making any type of casein. In the precipitating process soluble constituents of the skim milk are carried down with the curd, and unless the curd is washed thoroughly a considerable portion of the acid, soluble salts, and milk sugar present will be included in the final product.

Inadequate and ineffective washing of a casein curd lowers the quality of the finished product. Casein made from such curd is relatively high in free acid, sugar, and ash, and is often discolored during the drying process because of the presence of these substances. A high fat content in the skim milk may also cause discoloration during drying. Since large amounts of water are required in the washing of the curd, an adequate supply should be available. The acidity or alkalinity of the water should be determined by titration or with litmus paper and corrections made if necessary. The water pipe or hose lines to the vats should be sufficiently large—preferably 1¼ to 2 inches in diameter—so that the drained curd can be quickly covered with water. The sooner the curd is washed the better.

When the whey has drained from the curd, a quantity of wash water equal to that of the whey removed should be added to the curd, the mixture stirred throughly, the curd allowed to settle for a few minutes, and the water drained off. Unless otherwise specified, the temperatures of the wash water should be that of the whey, except that the last washing should be with cold water (about 60° F.) in order to cool the curd and retard acid formation in the press. All curds should be washed at least twice. Some manufacturers of the best quality casein wash the curd as many as six times. After the final washing, when the visible free water has drained off, the curd is ready to press.

In the continuous processes, washing is accomplished by sprays of water, the drainage being in a direction counter to that of the movement of the curd.

PRESSING

Pressing is necessary for curd of all types, with the exception of cooked curd. In batch pressing the washed, drained curd is usually shoveled from the vat into the press cloths. One of the cloths is placed over the press form, which is set on a drain board on the bottom of the press. Loose, wet curd is packed down into the cloth until the form is full, then the form is removed, and the cloth is lapped over the mass of curd. On this wrapped wet curd is placed another drain board, and the operation just described is repeated until all of the curd is stacked in the press. Care should be taken to have the curd stacked evenly, otherwise some of it may be pushed out of line when pressure is applied. With the ordinary screw-type, vertical press, the curd is pressed into solid cakes, about 30 inches square and 3 to 4 inches thick. When pressure is first applied a large amount of water is expelled. This is free moisture which contains small amounts of the soluble constituents of the skim milk. Pressing is, therefore, a refining as well as dehydrating process. The pressure should be increased at intervals. The usual factory practice of pressing overnight will produce solid cakes of curd that contain about 55 percent of moisture and will mill readily. Properly pressed curd yields a fine, mealy product when milled, one that will not become lumpy on the drying trays. If insufficient acidity was developed in the skim milk-whey mixture during precipitation, the curd will stick to the press cloths.

All press forms, drain boards, and press cloths, should be washed and scalded as soon as possible after being used, and the press cloths hung outdoors to dry.

When curd is pressed in a continuous press of the type illustrated in figure 2, one passage should reduce the moisture to about 55 percent

and give a cake that falls readily from the screen and is easily milled. Some types of continuous presses have several pairs of rolls, but one pair is as effective as more, provided the pressure is properly adjusted.

MILLING AND SPREADING

After the curd has been pressed overnight or passed through a continuous press, it should be milled and dried without delay, since moist curd will mold at ordinary temperatures. Unless the curd is of the cooked-curd type and hard or rubbery, it can be milled readily with a shredder-beater type of curd mill. The milled curd should be firm and mealy. It should be spread smoothly and evenly in a thin layer on the drying trays. Hand spreading is commonly practiced, though specially designed equipment is used in a few large factories. The trays should not be overloaded, as the drying will be retarded and the curd will tend to lump. About 2 to 2½ pounds of the finely milled curd to each standard tray 30 by 30 inches will give satisfactory results under general factory conditions. Care should be exercised in stacking the trays evenly on the tray trucks. The bottom screen on the truck should be of finer mesh than the others, or should be covered with a cloth, to catch fine particles of curd that may sift through the other screens.

DRYING

Control of the amount and humidity of the air used for drying, control of the circulation of the heated air over and through the moist casein, and careful regulation of the drying temperatures used, are essential in the drying of all caseins. The casein should be dried and not baked. In a tunnel drier with a drying temperature of 130° to 135° F., the heated air should be supplied at the rate of 4 cubic feet per minute per pound of wet curd per hour going into the drier. When conditions are properly controlled, a temperature of from 125° to 135° will dry the casein in from 6 to 8 hours. However, if the drier is inefficiently constructed or operated, from 12 to 16 hours may be required. Usually it is not advisable to operate the tunnel drier at temperatures below 125°. Temperatures as low as 110° to 115° will cause uneven drying and discoloration of the casein if conditions are not properly controlled. Temperatures higher than 135° should not be used at the beginning or near the end of the drying period. If the temperature at the start is too high, the outside of the particles dries first, a hard crust forming that hinders the escape of moisture from the interior. Thus the drying time is unnecessarily long and expensive and the quality of the casein is impaired. Near the end of the drying period evaporation becomes slower and the temperature of the tunnel will rise unless closely controlled. Too high a temperature at this stage will burn the casein, a condition manifest in a brown or reddish brown surface coloration. The drying process should be completed without interruption, and the heat should not be shut off before the casein is properly dried, as insufficient drying is favorable to growth of mold, and generally lessens the keeping properties of the casein.

A common factory arrangement for satisfactory drying is to have the fan located on top of the tunnel drier and preferably drawing

the fresh air from a point near the ceiling. The air is forced past the heater coils and through the tunnel, circulating between and around the loaded trays of moist casein, and becoming laden with moisture evaporated from the moist curd. The moist heated air is partly discharged through a flue to the outside of the building and partly recirculated, the amount recirculated through the drier being regulated by a damper arrangement or bypass. The dried casein preferably should not contain over 8 percent moisture, as a moisture content much higher than this adversely affects the keeping quality of the casein.

Continuous driers for casein are of several types. In the sectional, screen-conveyor drier, heated air is circulated up and down through the casein as it moves on a screen conveyor through three compartments, of which the first and last are kept at 130° F., and second at 200°. In the rotary drier, the casein travels slowly through a long, cylindrical, slightly inclined, rotating drum. The curd is continually being lifted on narrow shelves and dropped through a current of heated air moving counter to the travel of the curd. The temperature of the air at the end where the curd enters is kept at 120° to 130° and that at the discharge end at 160° to 170°.

GRINDING, PACKAGING, AND STORING

If casein from the drier is to be ground before being stored or shipped, it should first be thoroughly cooled in a clean bin, since warm casein will become gummy in a grinding mill. For producing a uniform product it is desirable to blend different batches of casein and this is most easily done by grinding them together. Most users of casein prefer it ground to pass a No. 20 or a No. 30 screen, but for some purposes a finer casein is demanded. Whatever degree of fineness is desired, the particles should be of as even a size as possible. To accomplish this, the mill should be adjusted for the size desired, never for a smaller size, and the casein screened after each grinding, the oversize particles being returned to the mill for regrinding.

The dried casein, either ground or unground, should be placed in clean dry sacks. Burlap sacks of 100 to 200 pounds capacity, lined with closely woven cloth or heavy paper, are satisfactory containers and are commonly used. One large manufacturer has adopted a heavy three-ply paper bag as a marketing package. This bag complies with all shipping requirements of the railroads and has been well received by the purchasers of the casein.

The filled sacks should be tightly closed and then put in a dry, clean, storage room where a uniform temperature can be maintained. Wide variations in storage temperatures will cause the stored casein to sweat and then mold. If the casein becomes sufficiently moist, decomposition will take place. Vermin may also infest stored casein. When it is known before grinding that casein is to be stored for any length of time, it is best to store it unground, because unground casein will keep better than ground casein.

In making different types of curd, as pressed curd, cooked curd, or grain curd, by batch processes, the principal differences necessary are in the precipitation equipment or type of vat used, and in the temperatures employed, the acidifying of the milk, completing the pre-

cipitation of the curd, washing, and finishing. As these operations have been discussed under general procedure they will be only briefly outlined here, with the kind of precipitation equipment necessary. Similar data are given for making continuous-process casein.

Pressed Curd

Casein of the pressed-curd type may be made by the use of comparatively inexpensive equipment, which, however, should include a drier, since wet pressed curd is not well adapted to shipping. A high-grade casein can be made by using either a self-sour or direct-precipitation procedure as here outlined.

SELF-SOUR

Precipitation equipment.—Vat with wooden rakes for stirring, or a mechanically stirred precipitating vat with a vat below for draining and washing.
Temperature of skim milk.—90° to 110° F.
Acidity.—Obtained by adding 5 to 10 percent of active starter and holding until the acidity is about 0.50 percent, or the pH value is 4.6 to 4.8.
Precipitation.—Completed either by allowing more acid to develop or by heating to not over 125° F. until whey is clear and curd is firm.
Washing.—The curd is settled, drained, and washed with cold water at least twice.
Finishing.—The washed curd is pressed, dried, and ground.

DIRECT PRECIPITATION

Precipitation equipment.—Same as for self-sour, pressed curd.
Temperature of skim milk.—90° to 110° F. with lactic acid, or 115° to 125° F. with sulfuric or hydrochloric acid.
Acidity.—For lactic type of curd, add sufficient high-acid whey (containing about 2 percent acid) to give an acidity of about 0.50 percent or a pH value of 4.6 to 4.8. For sulfuric type, prepare diluted sulfuric acid, consisting of one part of commercial acid added to six parts of cold water, and cool the mixture. **(Caution: Never add water to the acid.)** Slowly add dilute acid to the milk while stirring until a clean separation of curd and whey is obtained. This will be at about 0.35 to 0.40 percent acid. For hydrochloric, proceed as with sulfuric, except that the dilute acid is prepared by adding one part commercial acid to about three parts of water.
Precipitation.—Completed in the case of lactic curd by heating to 125° F. In making sulfuric or hydrochloric curd, sufficient acid is used to complete the precipitation.
Washing.—The curd is settled, drained, and washed with cold water two or three times.
Finishing.—The curd is pressed, dried, and ground.

Cooked Curd

The making of cooked curd requires only inexpensive equipment. No press is needed. The wet curd may be shipped to a centrally located plant for drying and grinding. Casein made by this process has poor solubility and is high in ash and free acid. This process therefore is not recommended.

SELF-SOUR

Precipitation equipment.—Vat fitted with direct steam.
Temperature of skim milk.—90° to 110° F.
Acidity.—Obtained by adding 5 to 10 percent of active starter and holding until acidity is about 0.50 percent or the pH is 4.6 to 4.8.

Precipitation.—Completed by heating with direct steam to 125° F. until whey is clear.

Cooking.—Whey is drained off, the curd is covered with warm water, and the water and curd are heated to 160° to 180° F. by direct steam until the curd is a rubberlike plastic mass.

Finishing.—The curd, while still warm, is packed in barrels for shipment, or, if the local equipment includes a drier, the curd is cooled, milled, dried, and ground.

DIRECT PRECIPITATION

Precipitation equipment.—Vat fitted with direct steam.

Temperature of skim milk.—120° to 125° F.

Acidity.—For the lactic type of curd add sufficient high-acid whey (containing about 2 percent acid) to give an acidity of about 0.50 percent or a pH value of 4.6 to 4.8. For the sulfuric type, use dilute sulfuric acid, consisting of one part commercial acid added to six parts of cold water, and cool the mixture. (Caution: Never add water to acid.) The dilute acid is slowly added to the milk with stirring, until a clean separation of curd and whey is obtained. This will be at an acidity of between 0.32 and 0.34 percent. For the hydrochloric type, proceed as with sulfuric, except that the dilute acid is prepared by adding one part of commercial acid to about three parts of water.

Precipitation.—Complete at temperatures and acidities given above.

Cooking.—The whey is drained off, the curd is covered with warm water, and the water and the curd heated to 160° to 180° F. by direct steam until the curd is a rubberlike plastic mass.

Finishing.—The curd, while still warm, is packed in barrels for shipment, or, if the local equipment includes a drier, the curd is cooled, milled, dried, and ground.

Grain Curd

The making of grain-curd casein requires means for close control of temperature and of pH value. A high-grade, low-ash casein can be made by this process, since the curd can be effectively washed. The hydrochloric acid type is the most readily washed.

HOLDING LACTIC

Precipitation equipment.—Vat with jacket to hold warm water.

Temperature of skim milk—At or near 100° F.

Acidity.—Obtained by the action of about 10 percent of an active, high-acid-producing starter. An acidity of from 0.55 to 0.65 percent should be obtained, preferably the latter.

Precipitation.—First stage: If acidity is about 0.55 percent and the curd soft, heat to 104° to 106° F. and stir until curd and whey have separated. If acidity is about 0.65 percent and the curd fairly firm, stir for from 10 to 15 minutes without increasing the temperature. The second procedure is the preferable one. Second stage: As soon as the curd has settled, as much of the whey is drained off as is quickly possible. Then, high-acid whey (containing about 2 percent acid) is added at 100° F. with stirring until the reaction is at pH 4.1. The curd will then be granular.

Washing.—The curd is settled, drained, and washed two or more times with acidulated water (pH 4.1) at 90° F., and once with cold water.

Finishing.—The washed curd is pressed, dried, and ground.

DIRECT PRECIPITATION

Precipitation equipment.—Vat with jacket to hold warm water.

Temperature of skim milk.—At or near 100° F. for lactic, 95° for hydrochloric, 98° for sulfuric.

Acidity.—pH 4.1, obtained by adding approximately 25 percent of sour whey containing about 2 percent of lactic acid; or by adding dilute hydrochloric acid, prepared by adding 1 part of commercial acid to 9 parts of water; or by adding dilute sulfuric acid, prepared by adding 1 part commercial acid to

at least 30 parts cold water. The skim milk should be slowly stirred during the addition of acid.

Precipitation.—Complete at pH 4.1 without further treatment.

Washing.—The curd is settled, drained, washed two or more times with acidulated water (pH 4.1) at 90° F., and once with cold water.

Finishing.—The washed curd is pressed, dried, and ground.

Continuous-Process Curd

The continuous process is used in localities where comparatively large quantities of skim milk are available the year round. The equipment is comparatively expensive. The casein produced is of the highest grade and uniform.

Equipment.—Continuous precipitator, such as that shown in figure 6, with continuous press. The drier may be of the tunnel or the continuous type.

Temperature of skim milk.—110° F.

Acidity.—pH 4.1, controlled by relative rate of flow of milk and acid into mixer. Dilute hydrochloric acid is most often used as the precipitant, prepared by adding one part commercial acid to three or four parts of water. Dilute sulfuric acid (1 to 10 or 1 to 20) or high-acid whey may also be used.

Precipitation.—Complete at pH 4.1; curd is stringy and coherent.

Draining.—Takes place as curd travels upwards on draining conveyor.

Washing.—After the drained curd passes through the curd breaker, it is washed by sprays of water as it travels upwards on the washing conveyor.

Finishing.—The washed curd passes through a continuous press, is milled, dried, and ground.

YIELD

The percentage of casein in skim milk is usually within a narrow range of values, the differences depending on such factors as the breed of the cows, the health of the cows, the lactation period, and the season of the year. Three percent is generally considered the amount present and, for statistical purposes, 35 pounds of skim milk is assumed to yield 1 pound of casein. The actual yield depends on details of the process of manufacture as well as on the quantity of casein present in the milk.

Inadequate washing of curd results in a yield of finished product greater than that obtained when the curd is washed effectively, since less casein is lost mechanically and more of the soluble impurities are left in the final product. A yield of 2.6 to 2.9 pounds of high-grade casein per 100 pounds of skim milk should be obtained from granular, well-washed curd. From coarse, lumpy, poorly washed curd, the yield may be 0.2 to 0.3 pound greater, but the casein will be of poorer grade. Any factor that influences the effectiveness of washing, such as, for example, the degree of dilution of the precipitating acid, the speed of stirring and foam removal, affects yield.

The maximum yield is desirable from the standpoint of having the maximum quantity of casein to sell, but maximum yield is obtainable only by the sacrifice of quality. Many plants could greatly improve the quality of their finished product, if the greatest yield consistent with best-quality casein were the objective, rather than the greatest yield regardless of quality.

COST OF MANUFACTURE

It is practically impossible to give exact figures on the cost of manufacturing casein. The costs of equipment and labor differ greatly

in different parts of the country, as does the value assigned to the skim milk. The quantity of milk processed is an important factor in determining costs per pound. Costs of from 2.49 to 8.23 cents per pound have been reported, with the average somewhat less than 5 cents. Excepting the cost of the skim milk, the largest single item is the cost of drying, which is 1 cent per pound, according to a number of manufacturers. This fact emphasizes the need for an efficient drier. Some of the direct items to be considered are the cost of water for washing the curd, of steam for heating the skim milk and for drying the curd, of power for operating the mechanical equipment, and of acid and labor. Indirect items include depreciation of equipment and building, interest, taxes, and insurance. Some factories have been able to decrease costs per pound of casein by purchasing skim milk from nearby creameries, thus increasing the volume of casein produced. With carefully designed, labor-saving equipment and a sufficient volume of skim milk, a plant should be able to produce dry casein at a cost of 3 cents per pound, exclusive of the cost of the skim milk.

USES

The uses of acid-precipitated casein depend chiefly on its adhesive and water-resistant properties. Over three-quarters of the casein consumed in this country is used in the coating of paper. Casein is used also in making glues and paints, especially cold-water paints, in insecticide sprays, in the textile industry as a filler, in finishing leather, and in the manufacture of linoleum.

For use, casein is first dissolved in an alkali solution, soda ash, trisodium phosphate, borax, and ammonia being the solvents commonly used in the paper-coating industry. Pigments such as clay and titanium oxide are added to the casein solution, which is then applied to the paper. The casein binds the pigments to the paper, thus producing a smooth, nonporous, opaque surface suitable for printing the finest books and magazines. Hydrated lime usually forms part of the solvent in casein glue and in casein paint because it helps to make casein water-resistant.

The commercial use of casein in making casein fiber began in the United States on a small scale in 1940. A carefully made, high-grade, acid-precipitated casein is necessary for making fiber, but the specially processed "textile" casein demanded by the manufacturers in Italy is not required for the domestic fiber.

MARKETING

The sales value of commercial casein is dependent upon the same general factors that govern the sales value of other manufactured products, and especially upon supply and demand, the physical and chemical properties desired, the uniformity of the product, the reliability of the manufacturer, the use to be made of the product, and its presentation to the trade in an attractive sales package. As the user becomes more appreciative of the value of high-grade casein, the manufacturer of such casein will have a big sales advantage over the manufacturer of an inferior casein. There seems to be a very definite trend on the part of certain large users of casein toward more exacting specifications. They are willing to pay a better price

for high-grade casein than for casein of inferior quality. This is a very encouraging condition, for too often in the past the buyer and the user have been interested primarily in the purchasing price, quality being only a secondary consideration.

As the value of casein depends so much on its physical and chemical properties, the manufacturer or prospective manufacturer should be familiar with them in order to be able to make a finished salable product which possesses as many of these desirable properties as possible.

Casein cannot be judged for quality merely by its appearance or by a physical examination. Exact methods are necessary to determine accurately the fitness of casein for the purpose intended. Some of these will be described later. Casein, however, possesses certain physical characteristics that can be used as an indication of whether the product has been properly made and handled. It should be uniform in color, clean, and free from objectionable odors and burned particles. The color should be a very light yellow or creamy white. The unground casein should be porous, crumbly in appearance, and not hard or glossy. A poor casein usually appears dull and often grayish in color, in contrast to the bright, even color of good casein. When ground it should be uniform in particle size and not dusty. The maker of casein should consider these indications of good quality in preparing his product for market.

Casein for most purposes should be clean, low in free-acid, fat, and ash, dissolve completely within certain limits of alkalinity and temperature, have good adhesive strength, and be free flowing when dissolved, and for paper coating spread easily without foaming. To determine accurately whether a casein is adapted to the purpose intended and the most equitable price that can be paid under the market conditions, actual utilization tests should be made. Then the manufacturer, to obtain the best price for his product in the future, should if necessary modify his manufacturing methods to supply casein that will comply with the user's requirements.

Casein is commonly sold through a dealer or jobber in carload lots. The small manufacturer is often handicapped by inadequate storage facilities and so must sell to a dealer. Such dealers have established contacts with the trade and are usually in a better position to market casein to advantage than is the individual manufacturer. From observations made in various sections of the country in 1931, it was found that the difference in prices paid the manufacturers ranged from 3 to 8 cents per pound. The manufacturer must be familiar with reliable current quotations in order to market his casein to best advantage.

Some manufacturers contract to sell their year's output at a definite price. Such an arrangement is satisfactory when the contract is elastic enough to protect both parties from taking undue advantage of each other. Casein prices fluctuate within rather wide ranges, and where a definite price is set for a year in advance one or the other of the contracting parties may find such an agreement undesirable. A flexible contract based upon market conditions would be fairer to both dealer and manufacturer.

Where the dealer is reliable and a fair price is paid, such a system of selling casein is probably more conducive to orderly marketing

than is an attempt on the part of a large number of small manufacturers to market direct to the user. As casein is usually sold in carload lots and a uniform product is desired, and the small factories use a variety of manufacturing methods, they are hardly in a position to supply such demands adequately. The popularity with the trade of much of the imported casein formerly used in this country was due not so much to any superior quality as to uniformity. This uniformity was due to a system of grading and blending previous to exportation. As uniformity is demanded by the user, the adoption of standardized manufacturing methods, together with a good system of grading, would seem to be highly desirable on the part of domestic manufacturers.

The large manufacturer of casein is in a much better position than the small one to market direct to the user. Usually in large plants the manufacturing methods are watched more closely and the product is more uniform than in small plants. Then too, a large producer is better able to establish good trade contacts. A familiarity with reliable market outlets should first be acquired, however, before direct marketing is attempted. A few large casein manufacturers have sales representatives to whom commissions are paid when sales are made.

New, clean sacks should be used. When more than one type of casein is made, each type should be appropriately labeled. The sales value of a good-quality casein, made by approved manufacturing methods and having the properties demanded by the user, will be increased if it is attractively packaged.

TESTING COMMERCIAL CASEIN

The following methods for testing casein have been selected for inclusion, partly because they give most readily an index to the degree to which casein possesses the properties desired by users, and partly because most of the tests can be carried out with fairly simple and readily available equipment. Details of other determinations, such as those of pH value and viscosity, may be obtained by writing to the Bureau of Dairy Industry, United States Department of Agriculture.

Sampling

Whatever the test being made, the sample used should be truly representative of the lot being tested. A sample of approximately a pound should be obtained by mixing together a few ounces taken from 8 or 10 different places in the lot. If not already ground, the sample should be ground to No. 20 size before mixing. Rolling the casein on a sheet of paper, first in one direction and then in another, is an effective method of mixing. The prepared sample should be placed in a tightly stoppered bottle.

Solubility

Solutions of borax in water are prepared containing 24, 30, and 40 gm. of borax ($Na_2B_4O_7.10H_2O$) per liter, and are labeled "12-percent

borax," "15-percent borax," and "20-percent borax," respectively. Fifty cubic centimeters of the 12-percent borax is added to 10 gm. of casein (passing a No. 20 screen), then the mixture is stirred and placed in a water bath at 150° F. (65° C.). It is stirred continuously for 5 minutes, and then again at the end of 10 minutes, and at the end of 15 minutes. If the casein is dissolved completely, it may be reported as "soluble in 12-percent borax"; otherwise the test should be repeated with another 10-gm. portion of the casein and 50 cubic centimeters of the next higher concentration of borax. If this portion is not entirely dissolved, a third portion is tried with the 20-percent borax. Only extremely poor caseins are insoluble in 20-percent borax. Frequently all the sample will dissolve except a few particles. These insoluble particles are usually attributed to foam on the milk during precipitation of the casein.

Adhesive Strength

Any test intended to show the strength of casein should approximate the conditions under which the casein is to be used. Formulas and techniques differ so much among users of casein that no one test will give information adequate for all users. Hence there follows the description of a test that will give comparative strength values for caseins that are to be used in coating paper. Some users of casein employ this test with refinements not described here, such as the use of a graded series of waxes to pull the coatings and close control of atmospheric conditions under which the tests are made. For most producers of casein, the test as described below will be sufficiently precise.

Twenty grams of casein are weighed into a previously tared beaker, which contains a stirring rod. About 70 cubic centimeters of water is added to the casein in the beaker, and its contents are allowed to stand with frequent stirring for half an hour. Then 2.4 grams of powdered chemically pure borax is added, and the mixture is warmed and stirred until solution is complete. Some caseins may require more borax to dissolve completely. It is essential that the mixture diluted with a few cubic centimeters of water shall turn an alcohol solution of phenolphthalein pink. When solution is completed enough water is added to make the total weight of the solution 100 grams. Each 5 grams of the solution then contains 1 gram of casein.

One hundred grams of dried clay is weighed into a casserole supplied with a blunt instrument for crushing lumps and mixing. A standard clay should be used. Kaolin, Special for Tannin, has been found satisfactory. Seventy cubic centimeters of water is added and the whole mixed to a smooth paste. The casserole and contents are then counterbalanced and 30 grams of the casein solution weighed in. After thorough mixing, a small amount of the mixture is spread on a sheet of paper by means of a straight-edged brass scraper or camel's-hair brush. The sheet is marked "6 grams," denoting the number of grams of casein, and put aside to dry. The casserole is again balanced, and 5 grams more of the casein solution is weighed in. The mixing and coating are repeated, and this sheet is marked "7 grams." Increments of 5 grams of the casein solution are added, and coatings are made up to "12 grams."

The coated papers are allowed to become thoroughly dry. Then, by holding them to the light, sections of uniform thickness are selected and marked. Sticks of good-quality sealing wax are well softened in a flame and pressed on the selected areas. Thirty minutes is allowed for the wax to cool. Then the fingers are placed on the paper beside the wax and the stick lifted with a steady pull. If the coating comes away without pulling any of the fibers of the paper, the test is negative. If the whole surface of the paper is pulled away, the test is positive. If part of the fibers are torn, the test may be considered as being on the border line.

High-grade caseins will give positive tests on the sheets marked "7 grams," "8 grams," or "9 grams." A casein which does not pull until the sheets marked "11 grams" or "12 grams" are reached is distinctly inferior in strength.

Since the result is affected by differences in the clay and in the paper used, it is important that the same grade of clay and type of paper be used consistently.

The quantities of clay, casein, etc., mentioned may be halved, if desired, provided the amount of mixture used for each separate coating is kept small.

Foaming Tendency

Foaming of clay-casein slips during application to paper is probably the most troublesome difficulty experienced in paper coating. The casein is nearly always blamed for this difficulty and usually properly so. The chief causes of this tendency in casein appear to be precipitation of the curd at temperatures below 90° F. and precipitation with too rapid agitation.

The following method for determining foaming tendency is essentially that devised by Richards and McFail,[3] and gives relative values. Different stirring assemblies will give different values on the same sample of casein.

A 110-volt 60-cycle alternating-current 1/15-horsepower synchronous motor mounted with shaft vertical and operating at a speed of 1,800 revolutions per minute, is used. The stirrer is mounted on the end of a round shaft one-fourth inch in diameter and 4 inches long, and consists of a blade 1⅜ inches long horizontally, one-fourth inch wide vertically, and one thirty-second of an inch thick. The stirrer and shaft are plated with nickel.

The slips are prepared by softening 11 gm. of casein in 55 cc. of water, adding one-half gram of soda ash, heating the solution to 50° C. and mixing with a suspension of 100 gm. of clay (kaolin, special for tannin) in 100 cc. of water. The dry clay is sifted through a 40-mesh sieve before being used.

One hundred cubic centimeters of the slip is poured into a 100-cc. graduate, and the graduate and its contents are weighed. Two hundred grams of slip is then placed in a 400-cc. low-form beaker, the motor-driven stirrer placed so that the top of the blade is three-fourths of an inch below the surface and the stirrer run for 5 minutes. After standing for 1 minute, 100 cc. of the slip is poured into the same 100-cc. graduate as before, and the graduate and the contents are

[3] Richards, C. W., and McFail, L. W. Development of method for determining foaming tendency of casein. Paper Trade Jour. 97 (1) : 29–30. 1933.

again weighed. The difference between the weights of 100 cc. before and after being stirred is a measure of the foaming tendency and is called the foaming index.

Moisture

Approximately 1 gm. of casein is weighed accurately into a tared silica or platinum dish and dried in a vacuum oven for 5 hours at 98° C. The sample is cooled in a desiccator and weighed, then dried 1 hour longer, cooled, and weighed. This operation is repeated until the weight is constant. The percentage of moisture is calculated from the loss of weight. The dry sample may be used for the determination of ash.

Ash

If the sample contains less than 2.5 percent of ash, the addition of calcium in the form of calcium acetate is necessary to prevent volatilization of organic phosphorus during the ashing process. Therefore, to prevent possible loss of phosphorus, 2 cc. of approximately tenth molal calcium acetate solution (equivalent to 0.0112 gm. of calcium oxide) should be added to the 1-gm. sample of casein, and the sample charred over a microburner without being allowed to burst into flame. The addition of 1 cc. of chemically pure nitric acid to the charred material is recommended as an aid in reducing it to ash. It is difficult to get a white ash without the addition of nitric acid. With some caseins it may be necessary to use 2 cc. of nitric acid. The acid is then evaporated over a low flame and the sample placed in a muffle, where it is heated at 600° to 650° C. until the ash is white. The exact weight of the calcium oxide added must be subtracted from the weight of the ashed sample before the percentage of ash is calculated. No correction is made for the nitric acid added, since it is volatilized completely during the heating.

Free Acid

Ten grams of 20-mesh casein is mixed thoroughly with 100 cc. of water, and the mixture is allowed to stand for an hour with occasional stirring. Fifty cubic centimeters of the supernatant solution is then titrated with tenth-normal sodium hydroxide. Phenolphthalein solution is used as the indicator. The solution should be stirred while it is being titrated, and the alkali slowly added until a faint pink color persists throughout the liquid. The free-acid value is expressed as the number of cubic centimeters of alkali solution required in the titration. An effectively washed casein will require less than 4 cc.

Fat

Several methods for the determination of fat in casein have been published. It is probable that the low values obtained by ether extraction of fat from solid casein are too low and it is known that substances other than fat are extracted by ether from solutions of casein. Hence, methods involving ether extraction are not recommended. The modified Babcock method here described is recom-

mended because it is rapid, apparently accurate, and can be carried out with equipment available in most dairy laboratories.

Two grams of casein is soaked in 6 cc. of water in a small beaker about one-half hour; then with constant stirring, 9 cc. of concentrated sulphuric acid (specific gravity 1.84) is added. This solution is poured into the Babcock skim-milk test bottle, the beaker is rinsed with 5 cc. of water and 5 cc. of sulphuric acid, and the washings poured into the test bottle. Then the bottle is filled to the base of the neck with dilute sulphuric acid (4 cc. of water and 5 cc. of concentrated acid) and whirled in the centrifuge for 5 minutes; filled to the top of the graduated portion of the neck with dilute acid, whirled 2 minutes, and the fat column read while still hot. The length of the fat column as read on the graduated neck, when multiplied by 9, gives the percentage of fat in the casein.

DEFECTS IN CASEIN AND THEIR PREVENTION

Poor Solubility

The cause of this defect may be (1) foam on the skim milk at the time of precipitation of the curd, (2) cooked curd, (3) moldy curd, or (4) the use of too high drying temperatures.

Preventive measures are (1) to remove foam from the skim milk before precipitating the curd, (2) to change the process to avoid cooking the curd, (3) to dry the curd promptly after making, and (4) to employ a lower drying temperature.

Dirty Casein

The causes may be (1) dirty plant equipment and surroundings or (2) dusty air entering the drier.

Preventive measures are (1) to clean all equipment of both loose and adherent dirt, to make floors or ceilings tight above equipment, to brush loose dirt from walls and floors, and (2) to make sure that the air intake for the drier is in as dirt-free a place as possible and screened.

Low Strength

The causes may be (1) a high percentage of ash or other impurities in the casein or (2) bacterial or mold action on the curd.

Preventive measures are (1) to make a more granular curd, to wash the curd a greater number of times, and (2) to press and dry curd immediately after washing.

High Ash

The causes may be (1) cooking of curd, (2) insufficient acidity during precipitation and washing of the curd, or (3) insufficient washing.

Preventive measures are (1) to make a more granular curd, using lower temperatures during the process of making, (2) to increase the acidity at the time of precipitation, to wash with acidulated water, and (3) to wash a greater number of times.

Foaming of Casein When Used in Paper-Coating

The causes of this fault attributable to the casein used may be (1) too low temperature during precipitation of the curd, (2) too rapid agitation during precipitation, or (3) other factors inherent in the milk.

Preventive measures are (1) to keep the temperature above 95° F. during precipitation, (2) to decrease the speed of agitation to barely enough to distribute the acid, and (3) to blend several lots of casein before shipment.

"Fish-Eyes" on Casein-Coated Paper

The causes may be (1) mold or other insoluble matter in the casein or (2) for some types of "fish-eyes," excess fat in the casein.

Preventive measures are (1) to press and dry the curd immediately after making (see also under Low Solubility and Dirty Casein), and (2) to improve the efficiency of the separation of cream from the skim milk used in making the casein.

Uncoated Spots on Casein-Coated Paper

The cause is practically always foaming of the coating mixture.

Preventive measures are the same as those for preventing foaming of coating mixtures containing casein.

LITERATURE ON COMMERCIAL CASEIN

More extensive information and a comprehensive bibliography of technical papers on casein and its uses have been published in a book by Sutermeister and Browne.[4] Of special interest to manufacturers of casein are the chapters on manufacture, analysis, and storage of casein.

[4] Sutermeister, E., and Browne, F. L. casein and its industrial applications. Ed. 2, 433 pp., illus. New York. 1939.

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